Fabrication and Properties of Graphene Oxide-Doped UO2 Pellets

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Abstract

Graphene Oxide (GO) doped uranium dioxide (UO_2) pellets represent a key research direction for high-performance nuclear fuels. To achieve uniform distribution of GO in UO_2 pellets and control over doping content, UO_2 powders with varying GO doping levels were prepared via solid-liquid mixing and ADU (Ammonium diuranate) co-precipitation methods, investigating the effects of powder preparation method and GO doping content on the mixing homogeneity of GO in UO_2. UO_2-GO pellets with different GO doping contents were fabricated by spark plasma sintering, exploring the influence of various sintering parameters, and the properties of the fuel pellets were evaluated. The results demonstrate that GO-doped UO_2 powders prepared by the solid-liquid mixing method exhibit superior mixing homogeneity; the density of UO_2-GO pellets obtained by spark plasma sintering can reach up to 97.6% T.D.; the thermal conductivity at 1000 ℃ of pellets with 1.5 wt.% GO doping content is increased by 85.9% compared to conventional UO_2 pellets; the prepared UO_2-GO pellets feature relatively uniform grain sizes, with GO uniformly distributed at grain boundaries and forming a bridging thermal conduction network, effectively enhancing the thermal conductivity of the doped pellets.

Full Text

Abstract

Graphene oxide (GO) doped uranium dioxide (UO_2) pellets represent a key research direction for high-performance nuclear fuels. To achieve uniform distribution of GO in UO_2 pellets and control the doping amount, UO_2 powders with different GO doping levels were prepared using solid-liquid mixing and ammonium diuranate (ADU) co-precipitation methods. The effects of powder preparation methods and GO doping amount on the mixing uniformity of GO in UO_2 were investigated. UO_2-GO composite fuel pellets with different GO doping amounts were fabricated by spark plasma sintering (SPS), the influence of various sintering parameters was explored, and the properties of the fuel pellets were tested. The results show that GO-doped UO_2 powders prepared by the solid-liquid mixing method exhibited better mixing uniformity. The density of UO_2-GO pellets obtained by SPS could reach up to 97.6% T.D. The thermal conductivity of pellets with 1.5 wt.% GO doping at 1000 °C was 85.9% higher than that of conventional UO_2 pellets. The fabricated UO_2-GO pellets exhibited relatively uniform grain size, with GO uniformly distributed at grain boundaries and forming a bridging thermal conduction network, which effectively enhanced the thermal conductivity of the doped pellets.

Keywords: Graphene oxide-doped modification, UO_2 pellet, Solid-liquid mixing method, Spark plasma sintering, Thermal conductivity

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Uranium dioxide (UO_2) fuel features a high melting point, excellent irradiation resistance, and good compatibility with cladding and coolant, making it widely used in current commercial nuclear power plants [1,2]. However, the low thermal conductivity of UO_2 fuel leads to steep temperature gradients within the fuel pellets during operation, resulting in significant thermal stresses that can generate cracks. These cracks accelerate fission gas release, while high fuel centerline temperatures exacerbate irradiation swelling, increasing reactor safety risks [2].

Doping UO_2 fuel with a second phase that possesses high thermal conductivity, high melting point, and favorable nuclear properties represents a key research direction for advanced nuclear fuels [3-7]. Graphene, composed of carbon atoms arranged in a single-layer sheet structure, exhibits excellent structural and chemical stability, with a theoretical thermal conductivity of 5000 $W/(m \cdot K)$, an elastic modulus of 1 TPa, and a tensile strength of 130 GPa, making it an ideal additive for improving the thermal conductivity and other properties of UO_2 fuel through doping modification [8,9].

In 2013, Korean researchers evaluated the in-reactor safety performance of UO_2-graphene composite fuel through computational analysis. The results indicated that 10 vol.% graphene doping could reduce the fuel centerline temperature from 2087 K to 1717 K, and decrease the cladding surface temperature during instantaneous startup from 1228 K to 1125 K, significantly increasing reactor power density and maximum allowable burnup [10].

However, research on graphene-doped UO_2 fuel remains in its early stages. Yao T et al. [11] fabricated graphene nanoplatelet (GNP) pellets with doping amounts of 1 wt.% and 5 wt.% using graphene nanoplatelet doping and spark plasma sintering (SPS), achieving thermal conductivities of 74% and 162% that of pure UO_2 pellets, respectively. Wu Xuezhi et al. [12] proposed an in-situ synthesis method where graphene served as nucleation sites for the reaction of uranyl nitrate solution with ammonia, depositing on these sites to prepare UO_2-graphene fuel powder doped with 2–8 vol.% graphene, with composite fuel pellets obtained through SPS. The study results showed that the thermal conductivity of the pellets could be improved by 58.93% at 1000 °C. Wang Y et al. [13] prepared UO_2-graphene nanosheet ceramic matrix composite fuel doped with 1, 3, and 5 wt.% graphene using mechanical mixing sintering and hot pressing sintering methods, achieving thermal conductivity improvements of 3.61%, 20.20%, and 45.67% at 700 °C, respectively. In previous studies, relatively large amounts of graphene doping yielded limited improvements in pellet thermal performance, and comprehensive characterization of overall pellet properties was insufficient, making it difficult to ensure that the intrinsic nuclear properties of UO_2 pellets were not compromised.

Research has shown that the comprehensive performance of UO_2-graphene composites is determined by the dispersion degree of graphene in the matrix and the quality of interfacial bonding. However, graphene exhibits poor wettability, making it difficult to form a good bonding interface with matrix materials, thereby limiting performance improvements of the composite [7,14]. Graphene oxide (GO) is a layered material produced by oxidizing graphite. Compared with pristine graphite, the graphene-derived flakes in GO are highly oxidized, containing hydroxyl and epoxide functional groups on their basal planes in addition to carbonyl and carboxyl groups located at the flake edges [15,16]. These functional groups render GO strongly hydrophilic, allowing it to easily swell and disperse in water, and consequently mix uniformly with UO_2 powder [8,17,18,14]. This study employed graphene oxide dispersion (0.5 wt.% GO content) as the primary doping raw material, with flake sizes smaller than 50 m. After strong acid oxidation, the increased oxygen-containing functional groups prevent agglomeration, facilitating uniform mixing with UO_2 powder.

SPS is a promising powder metallurgy sintering technology. In SPS sintering, sample powder is loaded into a mold made of graphite or other materials, and pressing pressure is applied to the powder using upper and lower mold punches and an electric arc [9]. By applying pressure, high-density compacts can be produced. Simultaneously, the electric field and pulsed direct current generate spark discharges at particle contact points, which can remove surface contaminants and adsorbates, thereby improving grain boundary diffusion processes [4,19]. Çelik Y et al. [20] investigated SPS technology for preparing graphene/Al_2O_3 composite ceramics and found that for samples with 3 vol.% graphene content, a relative density of 99.6% could be achieved when the SPS sintering temperature was 1350 °C.

The main challenge in preparing UO_2-graphene or UO_2-GO composite fuel lies in uniformly dispersing (graphene oxide) into UO_2 fuel and controlling its effective volume fraction in the pellets to maximize the excellent thermal properties of (graphene oxide) while minimizing damage to the intrinsic nuclear properties of UO_2. In this study, solid-liquid mixing and ammonium diuranate (ADU) co-precipitation methods were employed to prepare UO_2-GO powders with GO doping amounts of 0.5, 1.5, and 5.0 wt.%. After selecting the optimized powder preparation process, UO_2-GO composite fuel pellets were fabricated using the SPS method. The phase composition, density, thermal conductivity, and other characteristics of the UO_2-GO composite fuel pellets were examined and compared with conventional pure UO_2 pellets.

1.1 UO_2-GO Powder Preparation

The raw materials for powder preparation were GO suspension and depleted U_3O_8 powder. The GO suspension consisted of deionized water and 0.5 wt.% GO, with flake sizes smaller than 50 m. Uranyl nitrate solution was prepared using depleted U_3O_8 powder. Appropriate amounts of nitric acid and deionized water were added to a 2 L three-neck flask, which was then placed in an electric heating mantle. When the temperature reached 50 °C, mechanical stirring was initiated while a certain amount of depleted U_3O_8 powder was slowly added to the flask. After the U_3O_8 powder was completely dissolved, the mixture was filtered to obtain uranyl nitrate solution for subsequent powder preparation.

(1) ADU Co-precipitation Method
As shown in Figure 1 Figure 1: see original paper, GO solution was added to the uranyl nitrate solution and stirred for 30 min to ensure uniform mixing. Then, 150 mL of ammonia water (concentration 25%) was added to precipitate ADU ($(NH_4)_2U_2O_7$). After stirring, the mixture was aged for 30 min and filtered to obtain ADU filter cake containing GO. The ADU filter cake containing GO was dried in a muffle furnace at 350 °C for 2.5 h under N_2 atmosphere (99.9999%), then calcined at 650 °C for 30 min. The calcined ADU filter cake was reduced in H_2 (99.9999%) for 15 min, cooled to room temperature under N_2, and sieved. The GO addition amounts for different samples were 0.5 wt.%, 1.5 wt.%, and 5.0 wt.%, respectively. The blank sample (pure UO_2 powder) was prepared using a similar method but without GO addition.

(2) Solid-Liquid Mixing Method
As shown in Figure 1(b), the aforementioned pure UO_2 powder was slowly added to the GO solution, stirred for 30 minutes, and filtered to obtain UO_2-GO filter cake. The UO_2-GO filter cake was also placed in a muffle furnace and dried at 350 °C for 2.5 h under N_2 atmosphere, then heated to 650 °C for calcination for 30 min. The calcined material was reduced in H_2 (99.9999%) for 15 min. After the furnace temperature dropped to room temperature, the material was passed through a sieve to obtain UO_2-GO composite powder. The GO addition amounts for different samples were similarly 0.5 wt.%, 1.5 wt.%, and 5.0 wt.%, respectively.

1.2 Composite Fuel Pellet SPS Sintering

Appropriate amounts of UO_2-GO powder with different doping levels were placed into graphite molds and subjected to spark plasma sintering at 1880 °C and 35 MPa pressure for 30 min to obtain UO_2-GO composite fuel pellets. The specific temperature-pressure-time curve is shown in Figure 2 [Figure 2: see original paper].

1.3 Characterization and Testing Methods

A VEGA 3 XMU scanning electron microscope (SEM) and energy dispersive spectrometer (EDS) were used to observe the surface morphology of GO-doped UO_2 pellet samples. An AXIO Observer.Z1m automatic metallographic microscope was employed to examine the metallographic structure of the samples. The density of the samples was measured using the water immersion method with deionized water as the immersion medium. The carbon content in the samples was determined using high-frequency induction combustion-infrared absorption method with a CS600 carbon-sulfur analyzer, which has a detection range of 0.6×10^{-6} to 0.06 for a 1 g sample.

The specific heat of the samples was measured using a comprehensive thermal analyzer, and the thermal diffusivity was measured using a Netzsch LFA457 laser flash thermal conductivity instrument. Thermal conductivity reflects the heat transfer capability of a material, and in this study, thermal conductivity was determined as the product of specific heat, thermal diffusivity, and sample density.

2.1 GO Doping Uniformity and Content Control

In UO_2 pellets, higher GO mixing uniformity facilitates the formation of network bridge structures within the interior, which is beneficial for heat conduction from the inside outward. Therefore, the mixing uniformity of GO in the doped powder directly affects the thermal conductivity of the subsequent pellets. Samples were taken from different positions (top, middle, and bottom) of the prepared mixed powder (as shown in Figure 3 [Figure 3: see original paper]) to detect carbon content (i.e., GO content), and the coefficient of variation (CV) was used to evaluate the mixing uniformity [12]. The smaller the difference in carbon content among samples from different positions, the more uniform the powder mixture.

Let the carbon content measured each time be x_1, x_2, and x_3, respectively, then their average value is:

where s is the standard deviation and CV is the coefficient of variation. The mixing uniformity M can then be quantitatively expressed as M = 1 - CV%.

In the metallographic morphology of the samples shown in Figure 4 [Figure 4: see original paper], UO_2 grains can be clearly observed, with dark edges representing grain boundaries and GO. As can be seen from Table 1 and Figure 4, the mixing uniformity M of UO_2-GO powder samples prepared by the solid-liquid mixing method was generally superior to that of samples prepared by the ADU co-precipitation method, particularly when the GO doping amount was 1.5 wt.%, where the carbon content across different positions was more consistent. Wang Y et al. [13] demonstrated that using a similar mechanical mixing method, high-quality UO_2-GO powder could be obtained, and when the GO addition amount was 1 wt.%, the sintered samples achieved the highest density. In the following experiments, the solid-liquid mixing method was selected for UO_2-GO powder preparation.

Figure 5 [Figure 5: see original paper] shows the SEM morphology of powders prepared by the solid-liquid mixing method with different doping amounts. Flake-like graphene can be observed when the doping amounts are 1.5 wt.% and 5.0 wt.%.

2.2 SPS Sintering Parameter Optimization

Based on the research results in Section 2.1, UO_2 powder with a GO doping amount of 1.5 wt.% was selected for SPS sintering studies. Under inert atmosphere, the reaction between UO_2 and carbon begins at 1100 °C, becomes most vigorous around 1500 °C to produce UC, and then the reaction rate decreases rapidly [21]. The thermal conductivity of the reaction product is $21.7\ W \cdot m^{-1} \cdot K^{-1}$, which is 8 times higher than UO_2 but still significantly lower than that of GO. Therefore, the sintering temperature should be avoided around 1500 °C. The maximum sintering temperature was selected in the range of 1700–1950 °C, with reference to UO_2 sintering temperatures and heating rates. Considering pellet densification and minimizing reactions between graphene oxide and UO_2, the holding time was set to 10–30 minutes. The sintering pressure was determined based on the pressure-bearing limit of the graphite mold (4–15 kN). High-purity argon was used as the protective sintering atmosphere.

The actual sintering preparation scheme and test results are shown in Table 2 , with a maximum sintering temperature of 1700 °C, holding time of 10 min, and sintering pressure of 10 kN. It can be seen that when the heating rate was set to 100 °C/min before 1200 °C, 50 °C/min before reaching the maximum temperature, and the cooling rate was 100 °C/min, the obtained pellets exhibited intact appearance, relatively high density, and the best sintering effect. However, since the overall density of the pellets still did not meet design requirements, further optimization of the maximum sintering temperature, sintering pressure, and other parameters was necessary.

The density variation of pellet samples from SPS sintering can provide preliminary understanding of the sintering behavior of UO_2-GO composites. Figure 6 Figure 6: see original paper shows the effect of maximum sintering temperature on the relative density of pellets. It can be seen that the relative density of UO_2-GO pellets with different GO doping amounts could reach 90.0% T.D. above 1880 °C, but the further increase in sintering density was relatively small when the sintering temperature was further elevated. Therefore, the maximum sintering temperature was determined to be 1880 °C. GO doping inhibits sintering kinetics, as GO can act as a physical barrier during sintering, pinning atomic diffusion and grain boundary migration, thereby reducing the physical density and average grain size of the sintered body [11]. The results in Figure 6(a) and (b) indicate that longer sintering times or higher sintering temperatures promote sintering densification.

Studies have shown that the relative density of UO_2 is 96.1% at a sintering temperature of 1200 °C [22], whereas the relative densities of UO_2-GO samples with different doping amounts in Figure 6(a) at maximum sintering temperatures of 1700 °C and 1780 °C were all lower than that of the undoped blank sample. Since the sintering temperature of graphene is extremely high (above 2300 °C), it inhibits the densification process [23]. Compared with UO_2, UO_2-GO requires higher sintering temperatures to achieve densification. The density of UO_2-GO samples increased with increasing maximum sintering temperature. As the maximum sintering temperature increased, the UO_2 matrix became further densified, and pores gradually shrank and closed. At 1950 °C, the relative density was 91.3%, indicating that high-density interfacial products may have formed within the composite [21].

When the holding time was 20 minutes, the relative densities of undoped, 1.5 wt.% GO-doped, and 5.0 wt.% GO-doped samples were essentially consistent (Figure 6(b)), reaching 94.0% T.D. or higher. When the holding time was further increased, the densification degree of UO_2-GO pellets continued to increase, but the increment was relatively small. Considering both pellet density and GO retention, a holding time of 20 minutes was selected.

On the premise that the pellet density was sufficiently high (reaching 95.0% T.D.), the optimal sintering parameters for controlling carbon content were explored, as shown in Figure 6(c) and (d). Compared with the mixed powder shown in Table 1, the carbon content of sintered pellets increased significantly, primarily due to the volatilization of moisture and impurities during sintering. As the maximum sintering temperature of the pellets increased, the carbon content gradually decreased, and when the holding time increased from 20 min to 30 min, the carbon content dropped rapidly. Combined with the density of sintered pellets, when the maximum sintering temperature was 1880 °C and the holding time was less than 20 min, the effective carbon content of the pellets was optimally retained.

Additionally, based on the maximum pressure that the mold could withstand, the SPS sintering pressure could be increased to 15 kN. Under this pressure, the densities of undoped, 1.5 wt.% GO-doped, and 5.0 wt.% GO-doped samples could all reach above 96.1% T.D., with a maximum density of 97.6% T.D.

The microstructure of sintered pellet samples was observed using a metallographic microscope, as shown in Figure 7 [Figure 7: see original paper]. When the GO doping amount was 0.5 wt.% and 1.5 wt.%, the grain size was relatively uniform. As can be seen from the microstructure of UO_2-GO pellets in both Figure 4 and Figure 7, graphene oxide was mostly distributed at grain boundaries. Larger grain sizes can reduce heat transfer losses at grain boundaries, and the average grain size of UO_2-GO pellets with different GO doping amounts was generally larger than that of pure UO_2 pellets, indicating potentially better thermal conductivity.

2.3 Thermophysical Properties of UO_2-GO Pellets

The density and porosity measurement results of UO_2-GO pellet samples are shown in Table 3 . When the GO doping amount was 0.5 wt.%, the relative density was 93.4% T.D. When the GO doping amount was 1.5 wt.% or 5.0 wt.%, the relative density was greater than 94% T.D., meeting the design requirements for pellets. It can be seen that sample density did not always decrease with increasing GO doping amount. Additionally, when the GO doping amount was 1.5 wt.%, the porosity was 0.60%, indicating better densification of the pellets at this doping level.

The thermal conductivity of pellet samples sintered under five different parameter sets and with different GO doping amounts was measured, with results shown in Figure 8 [Figure 8: see original paper]. In this figure, – represent sample numbers, followed by parameters of GO doping amount (wt.%), maximum sintering temperature (°C), sintering pressure (kN), and holding time (min). The results indicate that the thermal conductivity of pellets with 1.5 wt.% GO doping was improved compared to both the 5.0 wt.% doped samples prepared with the same process and the blank samples, with a maximum improvement of approximately 86% in the high-temperature range (100–1000 °C) compared to blank samples.

In UO_2 ceramic materials, heat conduction is primarily achieved by phonons due to the lack of free electrons. Although phonons are sensitive to microstructural defects, they are more easily scattered and generally less efficient for heat conduction than free electrons. Figure 8(a) shows that the thermal conductivity of UO_2-GO samples generally decreases with increasing test temperature, which is mainly determined by the heat conduction mechanisms of UO_2 and GO. Heat conduction in both UO_2 and GO relies primarily on phonons, and as temperature increases, phonon scattering continuously increases, thereby reducing the thermal conductivity of UO_2-GO.

GO doping may exert two opposing effects on the thermal conductivity of UO_2 pellets. On the one hand, as mentioned previously, the thermal conductivity coefficient of graphene/GO is as high as $5300\ \mathrm{W} \cdot (\mathrm{m} \cdot \mathrm{K})^{-1}$, and GO doping is expected to improve the thermal conductivity of UO_2-GO composite pellets. On the other hand, some research results indicate that incorporating graphene into a ceramic matrix may instead reduce the material' s thermal conductivity. This is mainly because the introduction of graphene reduces sintering performance, leading to decreased density and increased porosity. Additionally, the thermal conductivity coefficient in the direction perpendicular to graphene flakes creates interfacial thermal resistance with the ceramic matrix, potentially reducing the overall thermal conductivity of the material. GO has a similar layered structure to graphene and thus may have similar effects. This explains why when the GO doping amount was increased to 5.0 wt.%, the thermal conductivity of the pellets decreased compared to when the GO doping amount was 1.5 wt.%.

Compared with Sample , Sample had a higher maximum sintering temperature but lower overall thermal conductivity. Excessively high sintering temperatures can affect the interfacial structure because the reaction between UO_2 and C becomes severe, and various crystalline phases may appear at the interface, creating new interfaces and increasing interfacial thermal resistance. Consequently, the thermal conductivity enhancement effect of GO is weakened [21]. However, compared with Sample , Sample showed a slight overall increase in thermal conductivity. The possible reason is that the maximum sintering temperatures of the two samples were not significantly different (only 20 °C apart), but the sintering pressure of Sample was significantly increased, which promoted sintering densification, improved sintering performance, and thus was beneficial for thermal conductivity improvement.

Conclusion

This work successfully prepared GO-doped UO_2 pellets, realized wet mixing via the solid-liquid mixing method, and significantly improved the distribution uniformity of GO in UO_2. By optimizing SPS sintering parameters including heating/cooling rates, sintering temperature, and holding time, effective retention of GO in sintered UO_2 pellets was achieved. Comprehensive out-of-pile performance comparisons were conducted with conventional UO_2 pellets for nuclear power applications, laying the foundation for subsequent in-reactor irradiation of this type of pellet.

This study investigated the effects of different GO doping amounts, powder preparation processes, and SPS sintering processes on the properties of UO_2-GO pellets, leading to the following conclusions: (1) According to metallographic examination and carbon content detection results in UO_2-GO powder, the solid-liquid mixing method can produce more uniform UO_2-GO powder compared with the ADU co-precipitation method, with a maximum powder uniformity of 99.16% and better mixed powder performance; (2) UO_2 pellets with different GO doping amounts were prepared by SPS sintering at 1780-1900 °C, and the density of UO_2-GO pellets could reach up to 97.6% T.D.; (3) When the GO doping amount was 1.5 wt.%, the thermal conductivity of UO_2-GO pellets was improved compared to both 5.0 wt.% GO-doped pellets prepared with the same process and undoped UO_2 pellets, with a maximum improvement of approximately 86% in the high-temperature range (below 1000 °C). GO was uniformly distributed at UO_2 grain boundaries and formed a bridging thermal conduction network, which can effectively improve the thermal conductivity of UO_2 pellets.

In future work, powder preparation and SPS sintering processes need to be further optimized to enable graphene or graphene oxide to form more three-dimensionally interconnected network structures in UO_2 pellets, further improving pellet thermal conductivity and providing superior material options for high-performance nuclear fuel optimization. Additionally, the UO_2-GO pellet preparation method proposed in this study can also increase preparation throughput based on optimized batch SPS sintering equipment to meet future large-scale application requirements.

Author Contributions: Wen Feng was responsible for experimental design and data analysis/interpretation; Du Jiangping conducted experiments and collected data; Zhao Yumeng drafted the manuscript and organized data; Yang Ning performed characterization tests and participated in manuscript revision; Shao Zongyi provided research ideas and theoretical guidance; Liu Wei provided resources and theoretical guidance; Cai Zhenfang provided administrative, technical, or material support.

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Note: Figure translations are in progress. See original paper for figures.